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IS 14437 (1997): Method for determination of quinalphos residues in agricultural and food commodities [FAD 1: Pesticides and Pesticides Residue Analysis]

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कृषि एवं खाद्य पदार्थों में क्वीनालफोस अवशेष ज्ञात करने की पद्धति

Indian Standard

METHOD FOR DETERMINATION OF QUINALPHOS RESIDUES IN AGRICULTURAL AND FOOD COMMODITIES

ICS 67.040

QBIS 1997

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9, BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

May **1997** **Price Group 2**

Pesticides Residue Analysis Sectional Committee, FAD 34

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Residue Analysis Sectional Committee had been approved by the Food and Agricultural Division Council.

Quinalphos formulations are extensively used in agriculture for the control of insect pests. This standard will enable the food, health authorities and others engaged in the field to follow uniform test procedure for the estimation of residues of quinalphos and its metabolities in various agricultural and food commodities.

In the preparation of this standard, due consideration has been given to the limits of quinalphos residues laid down under the provisions of Prevention of Food Adulteration Act, 1954 and Rules framed thereunder.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (revised)‘.

Indian Standard

METHOD FOR DETERMINATION OF QUINALPHOS RESIDUES IN, AGRICULTURAL AND FOOD COMMODITIES

1 SCOPE

This standard prescribes a gas chromatographic method for determination of residues of quinalphos [O,O-diethyl 0-quinoxalin-2-yl phosphorothioate], and its 2 metabolites, namely the P=O analog [o,o-diethyl o-(2-quinoxalinyl)-phosphate] and 2-hydroxyquinoxaline in agricultural and food commodities.

NOTE — This method has a detection limit lower than of 0.05 μg/g (0.05ppm) using the thermionic NP specific detector for all the 3 compounds and lower than 0.01 μg/g (0.01 ppm) using the alkali flame ionization detector for quinalphos and the P =0 along.

2 REFERENCES

The following standard contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standard indicated below:

IS No.	*Title*
11380 (Part 1) : 1985	Method of sampling for the determination of pesticide residues : Part 1 Agricultural and food commodities

3 SAMPLING

The representative samples for the purpose of estimating quinalphos residues shall be drawn in accordance with IS 11300 (Part 1) : 1985.

4 SAMPLE PREPARATION AND STORAGE

4.1 Sample Preparation

4.1.1 *Crops*

Grind the entire sample together with an equal weight of dry ice. Store the homogenized sample at −20°C in order to sublime the dry ice.

4.2 Sample Storage

Sample shall be stored as such when chances of its degradation do not exist and the quantity is small. Otherwise extraction shall be carried out and the extract stored. Depending upon the nature of the sample, keep the sample or its extract either in deepfreezer at − 15°C or in refrigerator until taken up for analysis. Ensure that the samples do not absorb or loose moisture during storage. Avoid undue long storage periods.

5 APPARATUS

5.1 Gas Chromatograph

The gas chromatograph shall be fitted with an alkali-flame ionization detector (AFID) and/or thermoionic NP specific detector (TID) and printer — plotter cum integrator. The following operating parameters are suggested, which may be varied provided standardization is done:

Column: Glass, 100 cm length x 2 mm I.D.: packed with 6 percent Carbowax 20 M on Gas Chrom Q (SO-100 mesh) or glass column, 200 cm x 2 mm I.D. packed with 0.2 percent Ultra-Bond 20 M Chromosorb W (SO-100 mesh) or glass column, 200 cm x 2 mm I.D. packed with 3 percent OV101/0V21 mixed on Chromosorb **WHP** (80-100 mesh) (for quinalphos and P = 0 analog only).

Temperature:

Column Oven

Carbowax 20 M - Gas Chrom Q	220°C
OV 101/OV 21 - Chromosorb WHP	200°C
Ultra-Bond 20M - Chromosorb W	190°C

Injection Port 250°C

Carrier gas and Flow rate: Nitrogen: 30 ml/minute

Air: 80 ml/minute

Fuel gas *and Flow rate:*	*40* ml/minute for quinalphos and P = 0 analog
	4.5 ml for Quinalphos, P = 0 analog and 2-hydroxly-quinoxaline

Detector AFID 330°C

(Quinalphos and P = 0 analog)

TID

250°C

(Quinalphos, P = 0 analog

2-hydroxlyquinoxaline)

5.2 **Microlitre** Syringe

10 μl.

5.3 Waring Blender or Equivalent

5.4 Centrifuge

5.5 Kuderna Danish Evaporative Concentrator or Equivalent

5.6 Sample Grinder

5.7 Mechanical Shaker

5.8 Screw-Cap Bottles

one litre capacity.

5.9 Chromatographic Column

Glass, 200 cm length x 25 mm I.D. with ground glass top.

5.10 **Buckner** Funnel

5.11 Rotary Vacuum Evaporator

5.12 **Extrelut** Column or Equivalent

150 mm length x 25 mm ID packed with 20 g extrelut material.

6 REAGENTS

6.1 Acetone

Glass re-distilled.

6.2 n-Hexane

Glass re-distilled.

6.3 Disopropyl Alcohol (Isopropanol)

Glass re-distilled.

6.4 Acetonitrile

Glass re-distilled.

6.5 Celite

545 or equivalent.

6.6 Dichloromethane

Glass re-distilled.

6.7 Toluene

Glass re-distilled.

6.8 Activated Silica Gel 60

Column chromatography grade (230-400 mesh), activated at 110°C for 6 h.

6.9 Sea Sand

Acid washed and purified.

6.10 Reference Standard Quinalphos

of known purity.

6.11 P = 0 Analog Reference Standard

of known purity.

6.12 **2-** Hydroxyquinoxaline Reference Standard

of known purity.

6.13 Reference Standard Solution of Quinalphos

Dissove 100 mg of reference standard quinalphos of known purity in toluene in a 10 ml volumetric flask and make up the volume to the mark.

6.14 P = 0 Analog Reference Standard Solution

Dissove 100 mg of reference standard P = 0 analog of known purity in toluene in a 10 ml volumetric flask and make up the volume to the mark.

6.15 **2-Hydroxyquinoxaline** Reference Standard Solution

Dissove 50 mg of 2-hydroxyquinoxaline reference standard of known purity in toluene in a 50 ml volumetric flask and make up the volume to the mark.

6.16 Filter Paper

Whatman No. 40 or equivalent

6.17 Hexadecane

7 EXTRACTION

7.1 Crops

Weigh 25 g homogenised sample into a screw-cap bottle of 1 000 ml capacity and add 10 g Celite-545 as filter aid and 100 ml acetone. Close the bottle and shake for 30 minutes in a mechanical shaker. If the crop material is not fine after grinding (about 1 mm), blend the sample together with 10 g Celite 545 and 150 ml acetone in a blender at high speed for 3 min and then shake in a mechanical shaker for 30 min. Filter through Whatman No. 40 or equivalent filter paper on a Buchner funnel, under suction. Return the residue to the screw-cap bottle, add 100 ml acetone and shake for 1.5 min. Filter as before, combining the filtrates. Evaporate the filtrate on in a rotary vacuum evaporator at 40°C under reduced pressure until only water remains in the flask.

NOTE- For dry crops, such as tea and straw, it is necessary to soak the sample in 10-20 ml water at ambient temperature overnight before extraction with acetone.

8 CLEAN-UP

8.1 Column Preparation

Place a plug of glass-wool at the bottom of the chromatographic column, which may withstand a pressure of about 2 atmosphere. Fill the column with 1 cm layer of acid washed and purified sea sand (see 6.9) followed by 10 g of silica gel and then another 1 cm layer of sea sand. Add 100 ml dichloromethane and press the solvent down through the column until its level is flush with the top layer of sea sand. This compression may be done by means of compressed air at a pressure of about 0.1 atmosphere. Repeat the process if necessary, until the silica gel is translucent (the dichloromethane eluent may be recycled).

8.2 **'Crops**

Transfer the aqueous extract from 7.1 into a 50 ml graduated droping funnel mounted over an Extrelut or equivalent column which in turn is mounted over the silica gel column (see Fig. 1). Wash the flask containing the extract with small amounts ofwater and transfer the washings into the dropping funnel until the total volume is 20 ml. Allow the solution to flow on to the Extrelut column and wait for about 10 minutes so that the solution is well absorbed on the whole column. Wash the flask further twice with 10 ml each of dichloromethane: hexane mixture (2: 1, *v/v*) and add the washings through the dropping funnel. Then add 200 ml of dichlormethane: hexane mixture and allow the eluate to come out from the silica gel column into a 500-ml round bottom flask. When the level of liquid reaches the top layer of acid washed and purified sea sand in the silica gel column, close the stop-coack. Remove the Extrelut column and add 100 ml of 20 percent isopropanol in hexane (*v/v*) to the silica gel column. Open the stop-cock and with the help of nitrogen at 0.1 atmospheric pressure, press the eluant through the column. Add 1 drop of hexadecane as keeper and evaporate to dryness in a rotary vacuum evaporator at 40°C under reduced pressure (30 to 50 mm of Hg). Dissolve the residue in toluene and quantitatively transfer to a 5 ml volumetric flask and make up the volume with toluene.

8.3 **Oils and Fats**

Dilute 15 g of oil or fat sample with 5 ml hexane and transfer quantitatively to the Extrelut column using 3 ml or more hexane for washing. After about 10 min, when the solution has been absorbed on the whole column, elute with 100 ml of acetonitrile and collect the elute in a 250-ml round bottom flask. Evaporate the elute to dryness in a rotary vacuum evaporator at 40°C under reduced pressure. Make sure that no acetonitrile is left in the flask. If it is found, then further clean up is necessary. Dissolve the residue in 10 ml dichloromethane andtransfer twice with using quantitatively on to the silica gel column twice with using each of 5 ml of dichloromethane: hexane mixture. Elute with 20 percent isopropanol in hexane (*v/v*) as described in 8.2.

9 ESTIMATION

Inject into the gas chromatograph, with the help of a microlitre syringe, a suitable aliquot of the mixture of reference standard solutions of quinalphos, the P = 0 analog and 2- hydroxygeninoxaline, identify the peaks by the retention times and measure the peak areas. Similarly inject an aliquot of sample extract and measure the peak areas of quinalphos, the P = 0 analog and 2-hydroxlyquinoxaline.

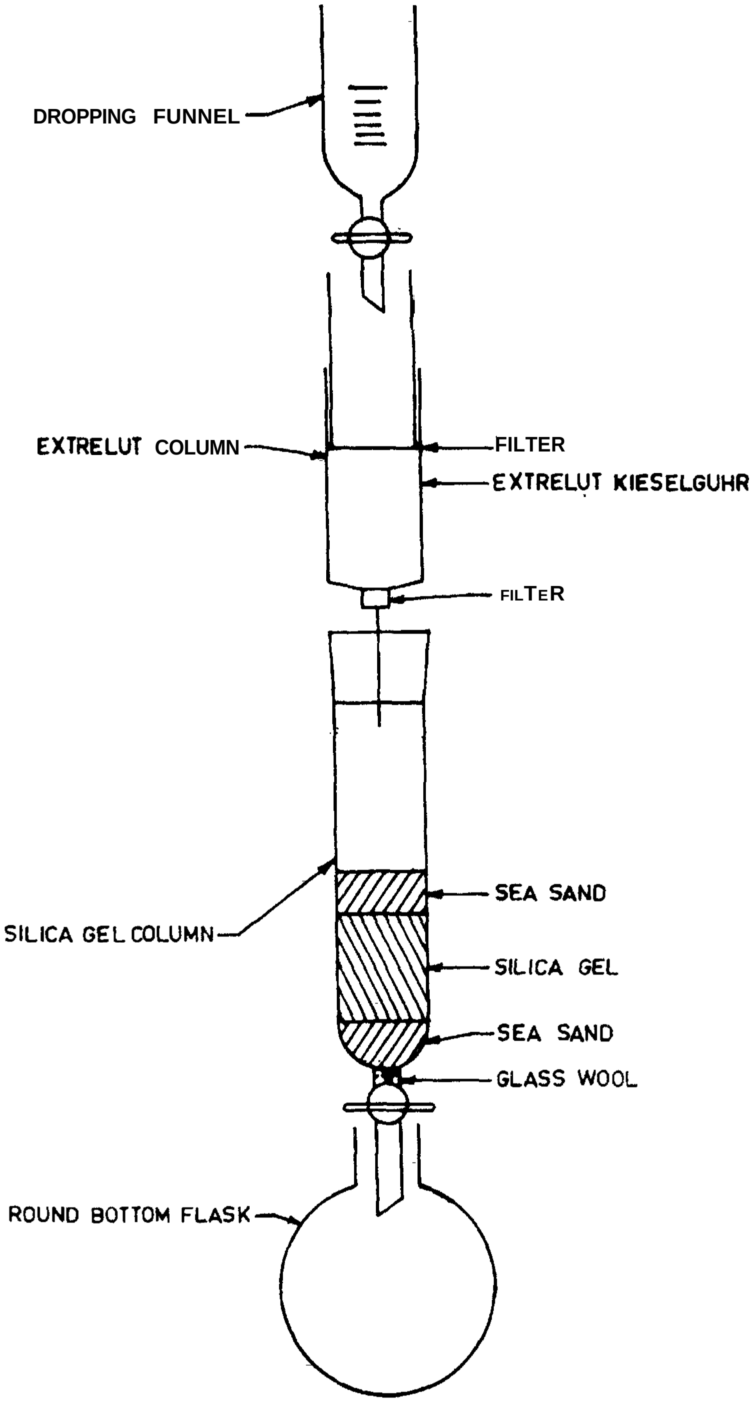

FIG. 1 APPARATUS

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